Examiner: Jonas N. Strickland

Group Art Unit: 1754



IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

pplicant:

Willard A. Cutler et al.

Serial No:

09/924,676

Filed:

08/08/2001

Title:

THERMALLY CONDUCTIVE HONEYCOMBS FOR

CHEMICAL REACTORS

Commissioner for Patents P.O. Box 1450 Alexandria, VA 22313-1450

Sir:

Transmitted herewith are three (3) copies of an Appeal Brief (8 pages with 2: page Appendix) in the above-identified application.

Authorization is given by Corning Incorporated to charge the appropriate fee and any additional fees necessary due in connection with this filing to Deposit Account No. 03-3325.

Dated: December 29, 2003

CERTIFICATE OF MAILING (37 CFR 1.8a)

I hereby certify that this correspondence is being deposited with the United States Postal Service with sufficient postage as first class mail in an envelope Addressed to: Commissioner for Patents, P.O. Box 1450, Alexandria, VA 22313=1450, on December 29, 2003.

Kees van der Sterre

Respectfully submitted,

CORNING INCORPORATED

Registration No. 25,938 Attorney for Assignee

(607) 974-3294

Corning Incorporated

Patent Department

SP-TI-03-01

Corning, NY 14831





IN THE UNITED STATES PATENT AND TRADEMARK OFFICE BEFORE THE BOARD OF PATENT APPEALS AND INTERFERENCES

Applicant(s): W. Cutler et al.

Appeal Brief

Serial No.:

09/924,676

Group Art Unit: 1754

Filing Date:

August 8, 2001

Examiner: Jonas N. Strickland

Title:

Thermally Conductive Honeycombs

for Chemical Reactors

Commissioner for Patents P.O. Box 1450 Alexandria, VA 22313-1450

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Klesryl Z Rees van der Sterre

This Appeal Brief is being filed in triplicate in support of the Notice of Appeal filed herein on October 27, 2003. It contains the following items under the corresponding headings as required by 37 CFR §1.192:

Real Party in Interest

Related Appeals and Interferences

Status of Claims

Status of Amendments

Summary of Invention

Issues

Grouping of Claims

Argument

Appendix (appealed claims)

Real Party in Interest

The real party in interest in this case is Corning Incorporated, assignee of the entire interest in this application by virtue of an assignment recorded 12/04/2001 at Reel/Frame 012341/0516.

Related Appeals and Interferences

There are no related appeals or interferences

Status of Claims

Claims 1-9 of the application stand finally rejected under 35 U.S.C. 102(b). Claims 10-14 of the application have been withdrawn as directed to a non-elected invention.

Status of Amendments

All amendments have been entered; no amendments have been refused entry.

Summary of the Invention

The invention relates to a method for making a metal honeycomb structure by the general process of extruding a metal-powder-containing extrusion batch through a honeycomb extrusion die, and to a metal honeycomb structure made by that method. The resulting honeycombs are useful as monolithic metallic substrates for the support of catalysts to be used in chemical reactors for carrying out exothermic or endothermic chemical reactions.

Important characteristics of such monolithic catalyst substrates include physical durability, good catalyst compatibility, and high thermal conductivity. High thermal conductivity is a property that is necessary to aid in the control of reaction temperatures within the catalyst beds formed by the monoliths and their supported catalysts.

An important aspect of the invention is an improved honeycomb forming process that includes steps to insure that metal oxides as well as organic binders and other organic constituents included within the metal-powder-containing extrusion batches are completely removed in the course of the process. Prior art processes for forming metal honeycombs from powders did not address this problem, and therefore frequently included residual oxide and/or carbon deposits that interfered with the catalyst support and/or heat transfer functions of the honeycombs. Those problems are more specifically detailed by the Applicants at page 3, lines 20-27 of the specification as follows:

For various reasons, these and other prior art extruded metal honeycombs have not yet proven satisfactory for use as catalyst substrates in many chemical reactions. Among deficiencies of the known extruded metal honeycombs are limited catalyst compatibility (due in part to excess carbon and other impurity levels), inadequate thermal conductivity for some reactions, a limited porosity range, and/or elastic properties that limit the physical durability of the substrates. On the other hand, metal honeycombs formed of aluminum or copper sheet stock demonstrate inadequate porosity, strength and durability for many catalyst support applications.

In accordance with the invention these problems are addressed through a postextrusion honeycomb consolidation process involving two discrete heating stages. In the first stage, the extruded honeycomb is heated in an oxidizing atmosphere at a temperature and for a time sufficient to remove organic binders and other extrusion aides and provide a carbon-free perform, and in a second stage the honeycomb is further heated in a reducing atmosphere for a time and at a temperature sufficient to sinter the carbon-free perform to an integral metal honeycomb (claim 1 of the application).

The resulting honeycombs provide physical durability as well as high thermal conductivity and good catalyst compatibility. They thus offer improved performance in selective oxidation reactions to make chemical products such as such as ethylene oxide, phthalic anhydride, maleic anhydride, formaldehyde, acrylonitrile, acrolein, acrylic acid, methacrolein, methacrylic acid,

methacrylonitrile, 1,2-dichloroethane, and vinyl chloride where temperature control is a critical process design consideration.

Issues

Whether the Examiner erred in finally rejecting claims 1-9 of the application as fully met by the disclosure of Harada et al. in EP 0450897 A2 (Harada).

Grouping of Claims

Claims 1, 3 and 4 are broadly directed to the Applicants' process as it would be applied to a selected set of thermally conductive metals including copper, steel, aluminum and the like.

Claims 2 and 5-9 are directed to thermally conductive metal honeycombs made of copper only.

Argument

The Examiner's rejection of claims 1-9 of the application under 35 U.S.C. §102(b) on reference to published European application EP 0450897 A2 to Harada et al. (Harada) clearly requires that all of the limitations of the claims be found in Harada. The Applicants respectfully submit that Harada clearly fails to disclose all of those limitations, and is therefore insufficient to anticipate the claims.

As noted above, all of the Applicants' claims require a two-stage heat treatment of a metal honeycomb perform formed by the extrusion of a metal powder batch in order to minimize or exclude of residual carbon and metal oxides from the structure of the finally consolidated metal honeycombs. The required two-stage treatment includes a preliminary heat treatment in an oxidizing atmosphere, and a secondary treatment in a reducing atmosphere as essential steps (page 5, lines 4-18 of the specification).

Harada's principal objective is to provide a heat-resistant, corrosion-resistant, and oxidation-resistant metallic honeycomb. The honeycombs thus provided are useful as catalyst supports and electric heaters (page 4, lines 25-31 of the reference). For that purpose thermal conductivity is irrelevant, and accordingly there is no reference to the problem of poor thermal conductivity or means for addressing that problem in the Harada disclosure. Instead what Harada requires is a metal honeycomb resistant to heat damage and thermal oxidation.

To provide these essential properties Harada expressly requires that both binder removal and sintering of the body be carried out in a <u>non</u>-oxidizing atmosphere (page 2, lines 10-15 and page 5, lines 52-55 of the reference). A preliminary heating step in an oxidizing atmosphere as the Applicants require is nowhere disclosed.

The Examiner cites only the Harada disclosure beginning at page 5, line 52 and continuing over to page 6, line 19 of the reference to support the view that the Applicants' two-stage oxidizing-reducing consolidation process is anticipated by Harada. That disclosure reads as follows:

Next, the shaped honeycomb body is sintered in a non-oxdizing atmosphere at a temperature ranging between 1000 and 1450°C. During the sintering in the non-oxidizing atmosphere containing hydrogen, the organic binder is decomposed and thereby removed with Fe or the like acting as a catalyst, and a good sintered body (a metal monolith) can therefore be obtained.

If the sintering temperature is lower than 1000°C, no sintering is achieved. Sintering conducted at a temperature higher than 1450°C causes deformation of the resulting sintered body or increases production cost and is therefore undesirable.

When a sinterable metal powder honeycomb monolith structure is to be sintered, the sinterable body is preferably encased in a sintering jig and thereby disposed close to or in contact with the jig. The sintering jig can be made of any material. Examples of such materials include metals, such as stainless steel, Mo and W, and ceramics, such as alumina, graphite and SiC.

The sintering time is appropriately determined such that the contents of C, N and O are within the above-described range. The preferred length of time is 2 hours or longer.

Thereafter, a heat-resistant metal oxide is preferably coated on the surface of the cell walls and that of the pores of the obtained sintered body by any of the following methods wherein:

- (1) the metal monolith (sintered material) is kept in a hydrogen-containing gas atmosphere of 500-1,300°C.
- (2) the metal monolith is subjected to the heat-treatment in an oxidizing atmosphere at a temperature ranging between 700 to 1200°C.
- (3) A ℓ or the like is plated on the surface of the cell walls and that of the pores of the sintered body (the metal monolith) (e.g., vapor plating) and that sintered body is subjected to the heat-treatment in an oxidizing atmosphere at a temperature between 700 and 1200°C.
- (4) the metal monolith is dipped into a molten metal, such as $A\ell$, and that metal monolith is subjected to the heat-treatment in an oxidizing atmosphere at a temperature between 700 and 1200°C.
- (5) alumina sol or the like is coated on the surface of the cell walls and that of the pores of the sintered body (the metal monolith) and that sintered body is subjected to the heat-treatment in an oxidizing atmosphere at a temperature between 700 and 1200°C.

Two conclusions are apparent from this disclosure. First, consolidation of the Harada honeycomb is to be carried out entirely in a non-oxidizing atmosphere. An initial heating step in an oxidizing atmosphere as required by the Applicants' claims is nowhere disclosed.

Secondly, the post-processing treatment to develop a metal oxide coating on the Harada honeycombs is carried out in an oxidizing atmosphere <u>after</u> the honeycombs have been consolidated. Even ignoring the fact that this second heat treatment occurs after consolidation has been completed, it apparent from the cited Harada disclosure that the sequence of a non-oxidizing heating step followed by an oxidizing heating step as in the reference is exactly opposite the oxidizing step followed by the reducing step required by the Applicants claims. Yet the Applicants' sequence is clearly critical in order to produce strong, catalyst-compatible, high thermal conductivity honeycombs in accordance with the claimed invention.

Given this reversal of the procedure expressly required by the Applicants' claims, it appears that there is no basis whatever to support a finding of anticipation of the Applicants' claims 1 and 2-4 by Harada. Accordingly, the Examiner's finding of anticipation by Harada under 35 U.S.C. §102(b) must be reversed.

Concerning the rejection of the Applicants' remaining claims 2 and 5-9 under 35 U.S.C. §102(b), those claims being directed to the method of producing thermally conductive copper honeycombs and to the products produced by that method, Harada again provides no express anticipation of the subject matter.

As Harada disclosure for fully meeting claims 2 and 5-9 of the application, the Examiner points only to the description at page 5, lines 31-32 of the reference. That disclosure is to the use of a minor optional copper addition to a honeycomb composition formed predominantly (at least 90% by weight) of Fe powder Cr power, and Al powder, as follows:

First, Fe powder, $A\ell$ powder and Cr powder, or powders of alloys of these metals, with optional additions of Sn powder and Cu powder, are mixed to prepare a metal powder mixture having a composition essentially consisting of, as analyzed in weight percent, 2. 5 to 30% $A\ell$, 0 to 40% Cr and a balance of Fe with the sum $A\ell$, Cr and Fe constituting 90% or more of the total composition.

It cannot be disputed that this teaching completely fails to provide a disclosure of a copper honeycomb, and there is no other teaching in Harada that does so. Whether the cited description is sufficient to suggest the removal of all other (and essential) AI, Cr and Fe components of the composition to yield a copper honeycomb (it does not) is clearly irrelevant. In the absence of an actual embodiment of a copper honeycomb in the Harada disclosure, the rejection of the Applicants' claims to a copper honeycomb under 35 U.S.C. §102(b) simply cannot stand. Thus the rejection of claims 2 and 5-9 as fully anticipated by Harada was also obvious error, and reversal of that rejection by the Board is therefore clearly required.

For all of the above reasons, reversal of the rejection of claims 1-9 of this application under 35 U.S.C. §102(b) on reference to Harada appears to be required under the facts of this case. Accordingly, reversal of that rejection and remand of this application to the Examiner for favorable reconsideration are respectfully solicited.

Respectfully submitted,

Kees van der Sterre Attorney for Appellants

Reg. No. 25,938

Corning Incorporated

SP-TI-03

Corning, NY 14831

(607) 974-3294

Date:December 29, 2003

Appendix - Appealed Claims:

- 1. A method for making a monolithic metallic catalyst substrate comprising the steps of:
 - compounding a metal powder extrusion batch comprising (i) a powder of a metal selected from the group consisting of copper, tin, zinc, aluminum, silver, iron, nickel, and mixtures and alloys thereof, and (ii) at least one carbon-containing temporary organic binder;
 - extruding the batch through a honeycomb extrusion die to form a honeycomb substrate preform;
 - heating the honeycomb substrate preform in an oxidizing atmosphere for a time and to a temperature at least sufficient to substantially remove the carbon-containing organic binder or extrusion aide by oxidation, thus to provide a carbon-free preform; and
 - heating the carbon-free preform in a reducing atmosphere for a time and to a temperature at least sufficient to sinter the carbon-free preform to a unitary monolithic metallic catalyst substrate.
- 2. A method in accordance with claim 1 wherein the powder is copper metal powder.
- 3. A method in accordance with claim 1 wherein the metal powder extrusion batch further includes an organic extrusion aide and a liquid vehicle.
- 4. A method in accordance with claim 1 wherein the carbon-free preform is heated in a reducing atmosphere at a time and for a temperature at least sufficient to achieve a honeycomb wall porosity in the range of 0-50% by volume.
- 5. A monolithic copper catalyst substrate produced in accordance with the method of claim 2.

- 6. A monolithic copper catalyst substrate in accordance with claim 5 which incorporates a high surface area washcoat and a metal or metal oxide catalyst.
- 7. A monolithic copper catalyst substrate in accordance with claim 6 wherein the washcoat is composed of alumina and the catalyst comprises a precious metal.
- 8. A monolithic copper catalyst substrate in accordance with claim 5 having a wall flow honeycomb configuration.
- 9. A monolithic copper catalyst substrate in accordance with claim 5 having a z-flow honeycomb configuration.



PATENT SP01-218

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE BEFORE THE BOARD OF PATENT APPEALS AND INTERFERENCES

Applicant(s): W. Cutler et al.

Appeal Brief

Serial No.:

09/924,676

Group Art Unit: 1754

Filing Date:

August 8, 2001

Examiner: Jonas N. Strickland

Title:

Thermally Conductive Honeycombs

for Chemical Reactors

Commissioner for Patents P.O. Box 1450 Alexandria, VA 22313-1450

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Kees van der Sterre

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Real Party in Interest

Related Appeals and Interferences

Status of Claims

Status of Amendments

Summary of Invention

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Argument

Appendix (appealed claims)

Real Party in Interest

The real party in interest in this case is Corning Incorporated, assignee of the entire interest in this application by virtue of an assignment recorded 12/04/2001 at Reel/Frame 012341/0516.

Related Appeals and Interferences

There are no related appeals or interferences

Status of Claims

Claims 1-9 of the application stand finally rejected under 35 U.S.C. 102(b). Claims 10-14 of the application have been withdrawn as directed to a non-elected invention.

Status of Amendments

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Summary of the Invention

The invention relates to a method for making a metal honeycomb structure by the general process of extruding a metal-powder-containing extrusion batch through a honeycomb extrusion die, and to a metal honeycomb structure made by that method. The resulting honeycombs are useful as monolithic metallic substrates for the support of catalysts to be used in chemical reactors for carrying out exothermic or endothermic chemical reactions.

Important characteristics of such monolithic catalyst substrates include physical durability, good catalyst compatibility, and high thermal conductivity. High thermal conductivity is a property that is necessary to aid in the control of reaction temperatures within the catalyst beds formed by the monoliths and their supported catalysts.

An important aspect of the invention is an improved honeycomb forming process that includes steps to insure that metal oxides as well as organic binders and other organic constituents included within the metal-powder-containing extrusion batches are completely removed in the course of the process. Prior art processes for forming metal honeycombs from powders did not address this problem, and therefore frequently included residual oxide and/or carbon deposits that interfered with the catalyst support and/or heat transfer functions of the honeycombs. Those problems are more specifically detailed by the Applicants at page 3, lines 20-27 of the specification as follows:

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In accordance with the invention these problems are addressed through a postextrusion honeycomb consolidation process involving two discrete heating stages. In the first stage, the extruded honeycomb is heated in an oxidizing atmosphere at a temperature and for a time sufficient to remove organic binders and other extrusion aides and provide a carbon-free perform, and in a second stage the honeycomb is further heated in a reducing atmosphere for a time and at a temperature sufficient to sinter the carbon-free perform to an integral metal honeycomb (claim 1 of the application).

The resulting honeycombs provide physical durability as well as high thermal conductivity and good catalyst compatibility. They thus offer improved performance in selective oxidation reactions to make chemical products such as such as ethylene oxide, phthalic anhydride, maleic anhydride, formaldehyde, acrylonitrile, acrolein, acrylic acid, methacrolein, methacrylic acid,

methacrylonitrile, 1,2-dichloroethane, and vinyl chloride where temperature control is a critical process design consideration.

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Whether the Examiner erred in finally rejecting claims 1-9 of the application as fully met by the disclosure of Harada et al. in EP 0450897 A2 (Harada).

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Claims 1, 3 and 4 are broadly directed to the Applicants' process as it would be applied to a selected set of thermally conductive metals including copper, steel, aluminum and the like.

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The Examiner's rejection of claims 1-9 of the application under 35 U.S.C. §102(b) on reference to published European application EP 0450897 A2 to Harada et al. (Harada) clearly requires that all of the limitations of the claims be found in Harada. The Applicants respectfully submit that Harada clearly fails to disclose all of those limitations, and is therefore insufficient to anticipate the claims.

As noted above, all of the Applicants' claims require a two-stage heat treatment of a metal honeycomb perform formed by the extrusion of a metal powder batch in order to minimize or exclude of residual carbon and metal oxides from the structure of the finally consolidated metal honeycombs. The required two-stage treatment includes a preliminary heat treatment in an oxidizing atmosphere, and a secondary treatment in a reducing atmosphere as essential steps (page 5, lines 4-18 of the specification).

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Next, the shaped honeycomb body is sintered in a non-oxdizing atmosphere at a temperature ranging between 1000 and 1450°C. During the sintering in the non-oxidizing atmosphere containing hydrogen, the organic binder is decomposed and thereby removed with Fe or the like acting as a catalyst, and a good sintered body (a metal monolith) can therefore be obtained.

If the sintering temperature is lower than 1000°C, no sintering is achieved. Sintering conducted at a temperature higher than 1450°C causes deformation of the resulting sintered body or increases production cost and is therefore undesirable.

When a sinterable metal powder honeycomb monolith structure is to be sintered, the sinterable body is preferably encased in a sintering jig and thereby disposed close to or in contact with the jig. The sintering jig can be made of any material. Examples of such materials include metals, such as stainless steel, Mo and W, and ceramics, such as alumina, graphite and SiC.

The sintering time is appropriately determined such that the contents of C, N and O are within the above-described range. The preferred length of time is 2 hours or longer.

Thereafter, a heat-resistant metal oxide is preferably coated on the surface of the cell walls and that of the pores of the obtained sintered body by any of the following methods wherein:

- (1) the metal monolith (sintered material) is kept in a hydrogen-containing gas atmosphere of 500-1,300°C.
- (2) the metal monolith is subjected to the heat-treatment in an oxidizing atmosphere at a temperature ranging between 700 to 1200°C.
- (3) A ℓ or the like is plated on the surface of the cell walls and that of the pores of the sintered body (the metal monolith) (e.g., vapor plating) and that sintered body is subjected to the heat-treatment in an oxidizing atmosphere at a temperature between 700 and 1200°C.
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Two conclusions are apparent from this disclosure. First, consolidation of the Harada honeycomb is to be carried out entirely in a non-oxidizing atmosphere. An initial heating step in an oxidizing atmosphere as required by the Applicants' claims is nowhere disclosed.

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Given this reversal of the procedure expressly required by the Applicants' claims, it appears that there is no basis whatever to support a finding of anticipation of the Applicants' claims 1 and 2-4 by Harada. Accordingly, the Examiner's finding of anticipation by Harada under 35 U.S.C. §102(b) must be reversed.

Concerning the rejection of the Applicants' remaining claims 2 and 5-9 under 35 U.S.C. §102(b), those claims being directed to the method of producing thermally conductive copper honeycombs and to the products produced by that method, Harada again provides no express anticipation of the subject matter.

As Harada disclosure for fully meeting claims 2 and 5-9 of the application, the Examiner points only to the description at page 5, lines 31-32 of the reference. That disclosure is to the use of a minor optional copper addition to a honeycomb composition formed predominantly (at least 90% by weight) of Fe powder Cr power, and Al powder, as follows:

First, Fe powder, A ℓ powder and Cr powder, or powders of alloys of these metals, with optional additions of Sn powder and Cu powder, are mixed to prepare a metal powder mixture having a composition essentially consisting of, as analyzed in weight percent, 2. 5 to 30% A ℓ , 0 to 40% Cr and a balance of Fe with the sum A ℓ , Cr and Fe constituting 90% or more of the total composition.

It cannot be disputed that this teaching completely fails to provide a disclosure of a copper honeycomb, and there is no other teaching in Harada that does so. Whether the cited description is sufficient to suggest the removal of all other (and essential) AI, Cr and Fe components of the composition to yield a copper honeycomb (it does not) is clearly irrelevant. In the absence of an actual embodiment of a copper honeycomb in the Harada disclosure, the rejection of the Applicants' claims to a copper honeycomb under 35 U.S.C. §102(b) simply cannot stand. Thus the rejection of claims 2 and 5-9 as fully anticipated by Harada was also obvious error, and reversal of that rejection by the Board is therefore clearly required.

For all of the above reasons, reversal of the rejection of claims 1-9 of this application under 35 U.S.C. §102(b) on reference to Harada appears to be required under the facts of this case. Accordingly, reversal of that rejection and remand of this application to the Examiner for favorable reconsideration are respectfully solicited.

Respectfully submitted,

Kees van der Sterre Attorney for Appellants

Reg. No. 25,938

Corning Incorporated

SP-TI-03

Corning, NY 14831 (607) 974-3294

Date:December 29, 2003

Appendix - Appealed Claims:

- 1. A method for making a monolithic metallic catalyst substrate comprising the steps of:
 - compounding a metal powder extrusion batch comprising (i) a powder of a metal selected from the group consisting of copper, tin, zinc, aluminum, silver, iron, nickel, and mixtures and alloys thereof, and (ii) at least one carbon-containing temporary organic binder;
 - extruding the batch through a honeycomb extrusion die to form a honeycomb substrate preform;
 - heating the honeycomb substrate preform in an oxidizing atmosphere for a time and to a temperature at least sufficient to substantially remove the carbon-containing organic binder or extrusion aide by oxidation, thus to provide a carbon-free preform; and

- heating the carbon-free preform in a reducing atmosphere for a time and to a temperature at least sufficient to sinter the carbon-free preform to a unitary monolithic metallic catalyst substrate.
- 2. A method in accordance with claim 1 wherein the powder is copper metal powder.
- 3. A method in accordance with claim 1 wherein the metal powder extrusion batch further includes an organic extrusion aide and a liquid vehicle.
- 4. A method in accordance with claim 1 wherein the carbon-free preform is heated in a reducing atmosphere at a time and for a temperature at least sufficient to achieve a honeycomb wall porosity in the range of 0-50% by volume.
- 5. A monolithic copper catalyst substrate produced in accordance with the method of claim 2.

- 6. A monolithic copper catalyst substrate in accordance with claim 5 which incorporates a high surface area washcoat and a metal or metal oxide catalyst.
- 7. A monolithic copper catalyst substrate in accordance with claim 6 wherein the washcoat is composed of alumina and the catalyst comprises a precious metal.
- 8. A monolithic copper catalyst substrate in accordance with claim 5 having a wall flow honeycomb configuration.
- 9. A monolithic copper catalyst substrate in accordance with claim 5 having a z-flow honeycomb configuration.